NASA Contract R-09-040-001

MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Quarterly Status Report
January 1, 1967 to April 1, 1967

U. S. Bureau of Mines NASA Program of Multidisciplinary Research
Leading to Utilization of Extraterrestrial Resources

QUARTERLY STATUS REPORT January 1, 1967 to April 1, 1967

Contents

STATUS REPORT THIRD QUARTER FISCAL YEAR 1967

U. S. Bureau of Mines NASA Program of Multidisciplinary Research Leading to Utilization of Extraterrestrial Resources

April 1, 1967

Task title: Core group activity
Investigator: Thomas C. Atchison, Senior Research Scientist
Location: Twin Cities Mining Research Center
Minneapolis-St. Paul, Minnesota
Date begun: April 1965 To be completed: Continuing

Personnel: Thomas C. Atchison, Supervisory Research Physicist
David E. Fogelson, Supervisory Research Geophysicist
Clifford W. Schultz, Research Extractive Metallurgist
Lowell W. Gibbs, Mining Methods Research Engineer
Other Bureau personnel, as assigned

PROGRESS REPORT

Objective

To provide the basic scientific and engineering knowledge needed for subsequent development of an extraterrestrial mineral resource extraction, processing and utilization technology for supporting and enhancing the economy of manned lunar and planetary missions.

Progress During the Third Quarter

The core group continued to obtain, evaluate, and distribute background information applicable to the program by literature search and direct contact with groups conducting related research. Members of the group attended the Fifth Annual Meeting of the Working Group on Extraterrestrial Resources (WGER) at the Marshall Space Flight Center in Huntsville, Ala., March 1-3, and the University of Michigan Symposium on Astrogeology in Ann Arbor, Mich., March 22-24.

While at Marshall we visited Hoyt Weathers and Stanley Fields at the Space Sciences Laboratory to discuss their studies of the effect of lunar vacuum on powdered rock materials. They are installing a new vacuum system with the same ultimate pressure capability as our systems but with a considerably larger chamber and faster pumping speed. There is a good possibility that we can work out a cooperative program with them to use their chamber for some of our experiments that might require more space or faster pumping speeds than our chambers can provide.

While at Ann Arbor we visited the Bendix Aerospace Systems Division and discussed their studies related to the Apollo lunar surface experiments package, the post-Apollo lunar surface vehicle, and infrared remote-sensing devices.

James Gangler, NASA manager for our program, has informed us that the proposal we submitted in December for continuing the work over the next two years has been approved and papers transferring an additional $300,000 to the Bureau are being prepared in NASA's Office of Research Contracts. We discussed with Mr. Gangler the possibility of additional funding for some of the present research tasks or for other tasks that we would like to add to the program. There are two possible sources of additional funds. One involves a new arrangement whereby a number of NASA offices have agreed to support specific studies relating to the use of extraterrestrial resources upon recommendation of the WGER. Proposals for such studies are to be submitted to the appropriate subgroup of the WGER for evaluation and selection for recommendation to NASA. A second source of funds is the Apollo Applications Program which involves a large number of earth orbital missions and a lesser number of lunar orbital and lunar surface missions over the next five to ten years. Funds are presently available for studies leading to specific experiments that can be carried out as a part of these missions. The type of experiments desired are those that will advance science or technology and which can be done more efficiently in space environment than in simulated environment on Earth. The core group will explore these two possibilities.

Status of Manuscripts

Mining on the Moon, by C. W. Schultz, was published in NEW SCIENTIST, July 9, 1966, p. 33.

Materials Testing Laboratory for Operation on the Lunar Surface, an informal report by Thomas C. Atchison, was submitted to NASA in August.

Objective and Method of Attack for Bureau Extraterrestrial Resource Utilization Program, by Thomas C. Atchison, and Scheduling of Research Tasks for Bureau Extraterrestrial Resource Utilization Program, by Lowell W. Gibbs, were presented at the NASA review meeting at the Twin Cities November 30.

Proposal for Continuing Bureau Extraterrestrial Resource Utilization Program, by the core group, was submitted to NASA in December.

Task title:	Selection and sample collection of simulated lunar materials
Investigator:	David E. Fogelson, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	September 1965 To be completed: Continuing
Personnel:	David E. Fogelson, Supervisory Research Geophysicist Other Bureau personnel, as assigned

PROGRESS REPORT

Objective

Select and obtain samples of rocks and minerals covering the range of materials likely to be found on the Moon.

Progress During the Third Quarter

As a result of an invitation from the program committee of the 1967 Pacific Northwest Metals and Minerals Conference, T. C. Atchison and D. E. Fogelson prepared a paper describing the purpose and progress of this task for presentation at the Conference. The theme of the Conference, to be held in Portland, Oreg., April 19-21, is "Materials for Inner and Outer Space."

At the Fifth Annual Meeting of the Working Group on Extraterrestrial Resources held at the Marshall Space Flight Center in Huntsville, Ala., Fogelson participated in the deliberations of the Environment and Resources Subgroup meeting chaired by Paul Lowman, Goddard Space Flight Center, Greenbelt, Md. The Subgroup agreed that major effort should be placed on developing practical methods that can be used during lunar orbital or lunar surface missions for identifying and evaluating potential resources. While at Huntsville we made arrangements with Hoyt Weathers of Marshall's Space Sciences Laboratory for a joint field trip to collect additional samples of our simulated lunar rocks in Oregon this spring.

At the University of Michigan Symposium on Astrogeology in Ann Arbor, Mich., Fogelson had the opportunity to discuss the Bureau's selection of simulated lunar materials with a number of geologists from universities and other research organizations from all parts of the United States.

Status of Manuscripts

Selection and Collection of Simulated Lunar Materials, by David E. Fogelson, was presented at the NASA review meeting at the Twin Cities November 30.

Simulated Materials for Lunar Mining Research, by Thomas C. Atchison and David E. Fogelson, was prepared and approved for presentation at the Pacific Northwest Metals and Minerals Conference, Portland, Oreg., April 19-21.

Task title: Physical properties of simulated lunar materials
Investigator: Thomas C. Atchison, Senior Research Scientist
Location: Twin Cities Mining Research Center
Minneapolis-St. Paul, Minnesota
Date begun: October 1965 To be completed: Continuing

Personnel: All projects are participating

PROGRESS REPORT

Objective

To incorporate simulated lunar materials into basic fragmentation research currently in progress. By this means to determine the composition, elastic, strength, surface, thermal, electrical, magnetic, and explosive shock properties of simulated lunar materials in Earth environment.

Progress During the Third Quarter

A number of property measurements were completed on the four rocks recently added to our simulated lunar materials. Density and porosity of the granodiorite, gabbro, and two additional vesicular basalts were determined by the Hydraulic Fragmentation group. An average pulse velocity was determined by the Rock Physics group. Uniaxial compressive and tensile strength, static Young's modulus, and Shore hardness were measured by the Mechanical Fragmentation group. Spheres of the rocks were prepared and added to the tests being carried out on all of the simulated lunar rocks to determine the effect of anisotropy on the strength and elastic properties.

Dissipation factor measurements over a frequency range from 20 to 100 megahertz were completed by the Thermal Fragmentation group on all of the rocks. Measurements of dielectric constant over the same frequency range were in progress and will be completed in the coming quarter.

Chemical and petrographic analyses of most of the rocks were completed during the quarter. Drillability data on four of the rocks in Earth environment were obtained by the Mechanical Fragmentation group as part of the drilling in lunar environment tasks. Plans were made by the Explosive Fragmentation group for a field trip to Oregon during the coming quarter to obtain explosive cratering data in a number of the rocks.

Preliminary studies of correlations among the different measured properties were made by the Thermal Fragmentation group as part of their analysis of data from tests on the effect of temperature on the modulus of rupture. Strength appears to correlate well with density when the rocks are separated into two groups, one including the surface volcanic rocks and the other the plutonic rocks. Further property correlation studies are in progress.

Table 1 gives preliminary average values for some of the properties on which measurements had been completed by the end of the quarter.

TABLE 1. - Properties of simulated lunar rocks in Earth environment

Rock type	Apparent density (g/cc)	Apparent porosity (percent)	Pulse velocity (m/sec)	Compressive strength (psi)	Tensile strength (psi)	Hardness (Shore units)
Dunite	3.19	1	7,500	27,000	2,000	73
Gabbro	3.11	<1	7,100	30,000	2,000	84
Flow basalt	2.84	2	6,000	53,000	3,400	84
Granodiorite	2.58	1	3,600	21,000	950	87
Serpentinite	2.56	3	6,000	18,000	800	68
Obsidian	2.39	1	5,600	65,000	2,200	103
Rhyolite	2.35	8	4,200	22,000	1,200	79
Vesicular basalt #1	2.25	20	3,800	10,000	1,100	81
Vesicular basalt #2	2.22	24	-----	5,500	590	67
Dacite	1.98	17	4,500	6,000	620	35
Vesicular basalt #3	1.52	46	4,800	5,600	810	80
Semiwelded tuff	1.15	50	2,500	850	100	10
Pumice	.76	62	2,500	1,500	240	5

Status of Manuscripts

None in progress.

Task title:	(1) Chemical reactivity and cold welding of freshly formed surfaces (2) Surface properties of rock in lunar environment
Investigator:	Clifford W. Schultz, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	January 1966 To be completed: March 1969
Personnel:	Clifford W. Schultz, Research Extractive Metallurgist William H. Engelmann, Research Chemist Wallace W. Roepke, Physical Science Technician Kenneth G. Pung, Physical Science Technician Ernest Bukofzer, Engineering Technician

PROGRESS REPORT

Objective

Measure the equilibrium constants for the adsorption of gases on the surfaces of silicate minerals. Relate this quantity to the fractional coverage necessary to inhibit cold welding and determine the rate at which various other processes inhibit or prohibit cold welding of vacuum formed surfaces. Extend current experimental studies of surface properties of rocks and minerals to include lunar environment.

Progress During the Third Quarter

The measurements of the equilibrium constants for the adsorption of water vapor on silicate minerals were deferred to the fourth quarter. During the past quarter components for a gas cell for the infrared spectrophotometer were purchased, assembled and installed. The IR spectrophotometer will be used for gas analysis in place of the thermal conductivity cell which was found to be inadequate. Calibration of the IR unit is now in progress.

The Ultek Quadrapole mass spectrometer has been received, checked, and accepted. Prior to acceptance of the unit, Mr. James Wolf of Ultek spent two days here checking the instrument and instructing Messrs. Roepke, Engelmann and Pung in its maintenance and operation.

The mass spectrometer has been used for the balance of the quarter on the NRC oil-pumped system and the Ultek ion-pumped system to study residual gases under various operating conditions. The hydrocarbon background in the oil-pumped system was found to be so high that further outgassing studies on the simulated lunar standards are being done exclusively in the ion-pumped system.

Initial attempts to study outgassing rates in the ion-pumped system with an elastomer seal (Viton) were deemed unsatisfactory because of the unusually high nitrogen background. Additional tests have been run using copper crush seals. Data from the latter tests showed an improvement in system performance. Figures 1 and 2 show the behavior of basalt, and semiwelded tuff in the system as compared with the empty system. The bakeout and pumpdown cycle is shown in figure 1. Although continuous pumping is used the system pulls down too fast to show the pressure above the 5 x 10^{-6} torr range. The sharp break in the curves indicate a sudden decrease in outgassing as the bakeout heaters go off. The rate of rise curves shown in figure 2 are obtained by switching off the pump and observing the pressure rise in the closed system. These data confirm earlier indications from the oil-pumped system. Tentatively it may be said that:

1. The tuff with its higher surface area presents a greater total gas load to the system.

2. The tuff, being extremely porous, approaches a state of equilibrium with the system during pumpdown.

3. The basalt outgasses very slowly indicating that diffusion is the rate controlling mechanism.

4. Both samples preferentially adsorb water. This is indicated by the absence of water in the background spectra, except during periods of bakeout.

In future tests, an attempt will be made to avoid bolting the chamber each time by replacing the copper seal with indium. This material has a very low vapor pressure and is softer than Viton. A considerable saving in time will be realized if the indium provides a seal without bolting.

Status of Manuscripts

Chemical Reactivity and Cold Welding - Surface Properties, by C. W. Schultz, was presented at the NASA review meeting at the Twin Cities November 30.

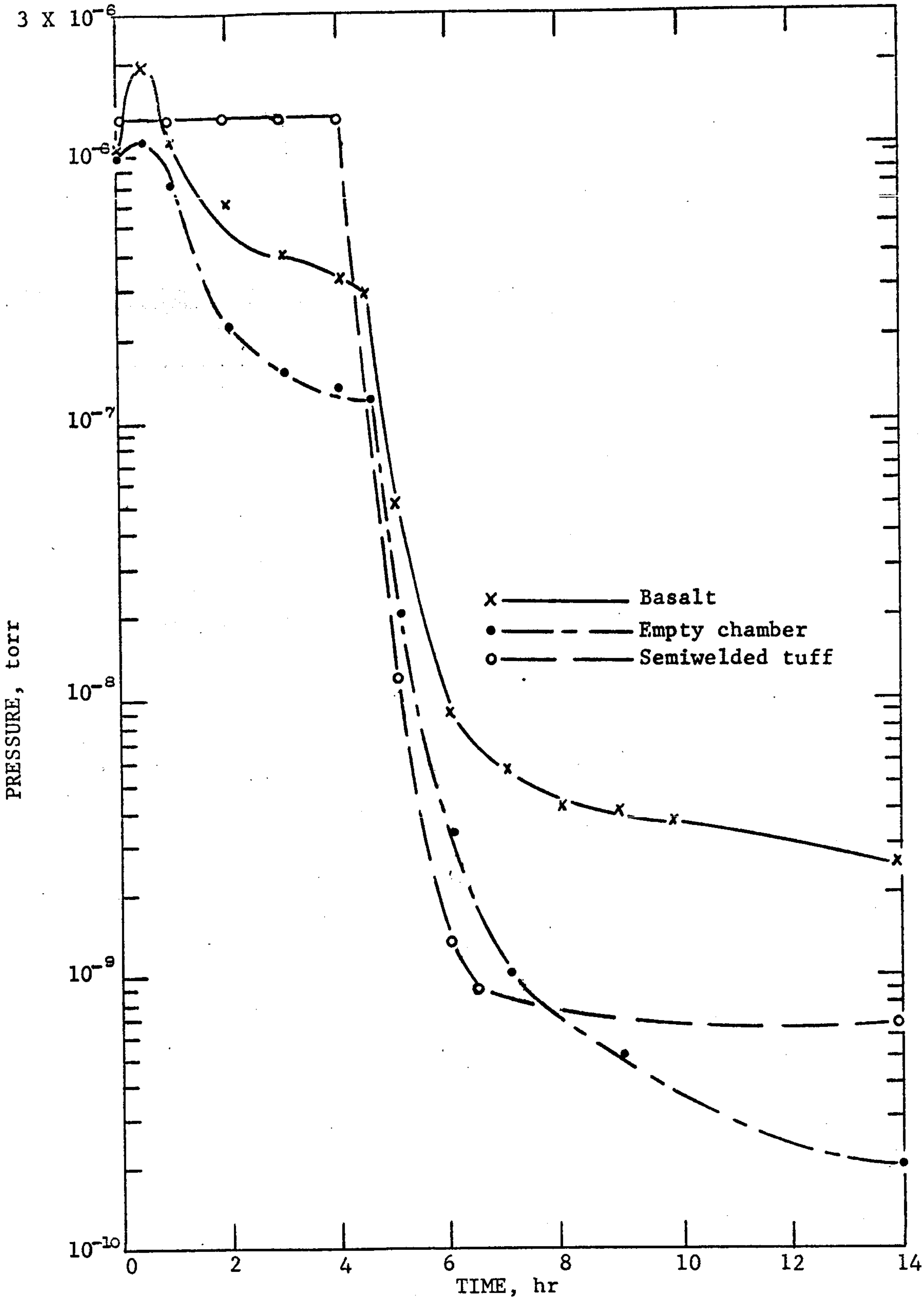

FIGURE 1. - Pumpdown Rates for Basalt, Semiwelded Tuff, and an Empty Chamber.

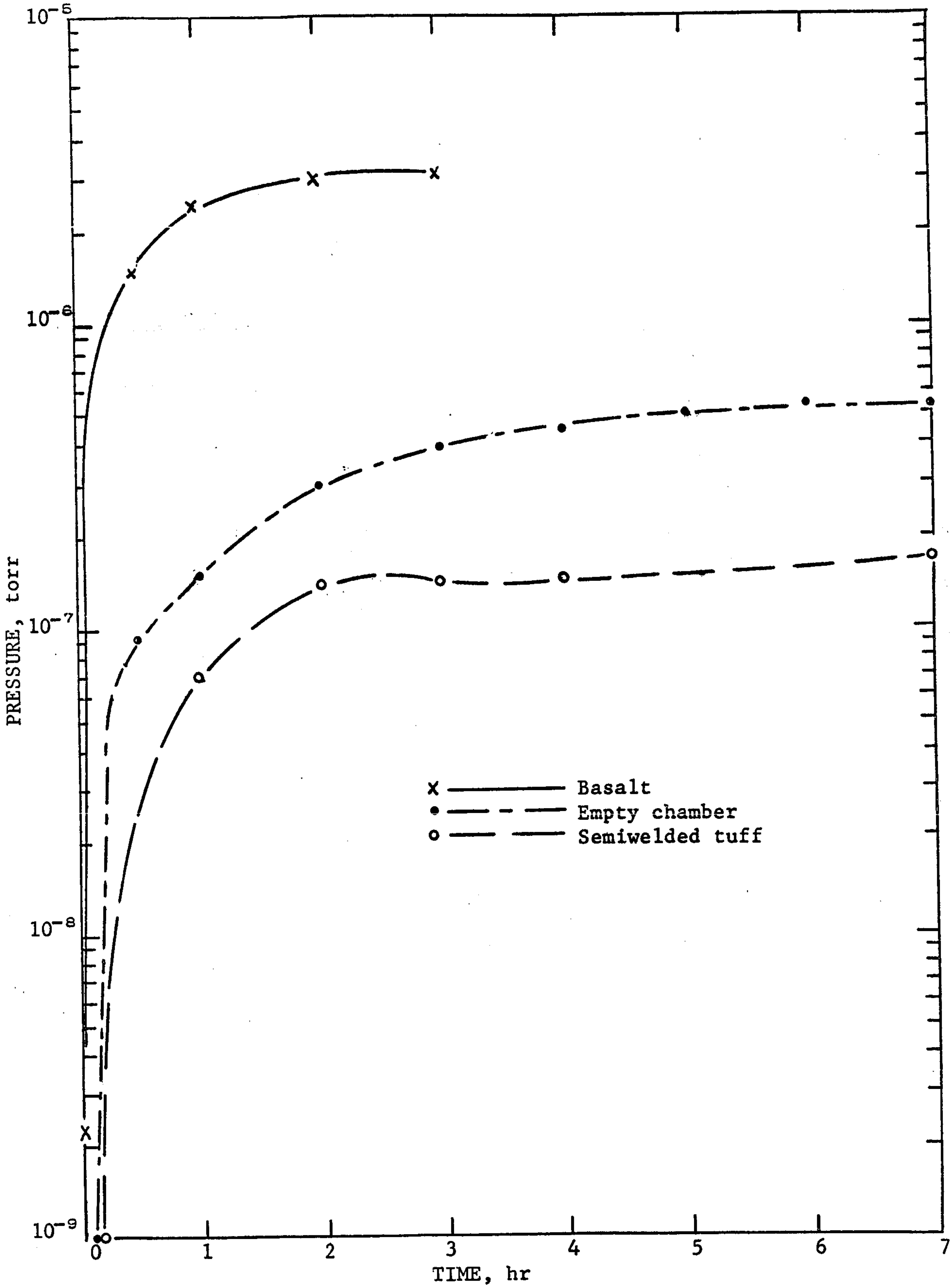

FIGURE 2. - Rate of Pressure Rise for Basalt, Semiwelded Tuff, and the Empty Chamber.

Task title:	(1) Fracture and other failure mechanisms in lunar environment (2) Strength and elastic properties of rock in lunar environment
Investigator:	John R. McWilliams, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	June 1966 To be completed: June 1969
Personnel:	John R. McWilliams, Supervising Mining Research Engineer Robert J. Willard, Geologist Thomas R. Bur, Research Geophysicist Egons R. Podnieks, Mechanical Research Engineer Richard E. Thill, Geophysicist Peter G. Chamberlain, Geophysicist Kenneth E. Hjelmstad, Geologist Richard M. Brumley, Electronics Technician

PROGRESS REPORT

Objective

Extend current experimental studies of rock failure by such mechanisms as dislocation, twinning, and crack formation to include lunar environment. Extend current measurements of static and dynamic elastic moduli and compressive and tensile strengths of rock to include lunar eniironment.

Progress During the Third Quarter

The study of the effect of environment on the static and dynamic properties of four rocks (basalt, dacite, granite, and limestone) continued on schedule. A major portion of the test matrix has been completed including Young's modulus, sonic velocity, point load tensile strength and uniaxial compressive strength measurements for all four rock types under the following environmental conditions: dry, 50 percent relative humidity, and 100 percent relative humidity, at room temperature at 212°F. During the fourth quarter, the test matrix will be completed by extending the above measurements to include liquid nitrogen temperature and moderate vacuum.

Preparations for measurements at lunar vacuum continued with modifications and adaption of the Ultek ultrahigh vacuum system for use with our testing system. The major problems solved during the quarter were relocation of the sorption pumps, the design of the bellows-supported platens for in vacuo physical property tests, and the design of the supporting structure to move the chamber into and out of the testing machine.

Status of Manuscripts

Rock Failure Mechanisms - Strength and Elastic Properties, by J. R. McWilliams and R. J. Willard, was presented at the NASA review meeting at the Twin Cities November 30.

Task title:	(1) Rock vaporization, melting, and thermal fracturing methods in vacuum (2) Thermophysical, strength, and elastic properties of rock at elevated and reduced temperatures in vacuum
Investigator:	Robert L. Marovelli, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	October 1966 To be completed: September 1968

Personnel:	Robert L. Marovelli, Supervising Mining Research Engineer Russell E. Griffin, Electronic Research Engineer Ta-Shen Chen, Mechanical Research Engineer Carl F. Wingquist, Physicist David P. Lindroth, Physicist Sam G. Demou, Physicist Daryl J. Jersak, Engineering Technician Walter G. Krawza, Engineering Technician

PROGRESS REPORT

Objective

Investigate the feasibility of extending current thermal fragmentation studies to lunar vacuum environment. Currently the thermophysical, strength, and elastic properties of rock at temperatures up to the melting point are being measured. Extend this work to the low temperature range of lunar environment. Investigate the feasibility of extending these property measurements to lunar vacuum environment.

Progress During the Third Quarter

Studies of the effect of temperature on the strength of rock were continued. Modulus of rupture (bending strength) data are being obtained on the complete suite of simulated lunar rocks over the lunar temperature range. Test specimens of 13 rock types were prepared and split with the University of Wisconsin personnel at Madison. Tests at -320°F and 75°F are in progress at Madison; tests at 75°F and 600°F are in progress at Minneapolis.

A laboratory experiment is in progress to determine the effect of moisture and stress state on thermal spalling of rock. Heat flux and exposure time are being held constant. Quartzite, granite, and basalt are being spalled and compared: after an air dry, after a 30-day water soak, after a bake at 430°F in vacuum, after a bake at 600°F in air. Similarily treated specimens will be spalled while under mechanically induced stress.

Dr. Chen completed a study of thermal spalling and left the Center to teach thermodynamics and heat transfer at the University of Missouri.

He remains available for part-time work. Chen performed an analysis based on (1) the uncoupled quasi-static theory and (2) the uncoupled theory of thermoelasticity. He used a stress point of view and worked with a semi-infinite body as a model. Experimental work showed disagreement between experimental and theoretical results. Chen concluded that the thermal spalling phenomena cannot be satisfactorily explained solely from knowledge of the stress distribution. He recommended that consideration of strain energy and the coupling of mechanical and thermal energy equations be tried next.

Melting and thermal shock experiments on the simulated lunar rocks have been delayed by the late delivery of the thermal shock furnace. This equipment was finally received and installed during the quarter. All components, including the vacuum system providing a 10^{-5} torr capability, have been checked out and are operating satisfactorily.

Status of Manuscripts

Thermal Fragmentation - Properties at High and Low Temperatures, by R. L. Marovelli, was presented at the NASA review meeting at the Twin Cities November 30.

The Effect of Low Temperatures on Some Physical Properties of Rock, by R. W. Heins and T. O. Friz, was presented at the University of Texas/AIME Drilling and Rock Mechanics Conference in Austin, Texas on January 26.

Flexural Strength of Rock from -320° to 1600°F, by R. L. Marovelli and A. Hendrickson, is under preparation as a journal article and a Bureau of Mines Report of Investigations.

Task title:	(1) Cuttings removal in drilling in lunar environment (2) Cooling and lubricating bits in drilling in lunar environment
Investigator:	James Paone, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis-St. Paul, Minnesota
Date begun:	January 1967 To be completed: December 1969
Personnel:	James Paone, Mining Methods Research Engineer Dick L. Madson, Mining Methods Research Engineer Robert L. Schmidt, Mining Engineer Vacancy, Physicist Vacancy, Electronic Development Technician David A. Larson, Engineering Technician

PROGRESS REPORT

Objective

Investigate various means of removing drill cuttings with and without flushing media in lunar environment. Investigate problems of heat removal and bit lubrication associated with drilling in lunar environment.

Progress During the Third Quarter

Laboratory drilling tests were begun using simulated lunar rocks. Flow basalt, vesicular basalt, rhyolite, and dacite were selected to represent a range of physical properties.

A sample of each material was drilled in atmosphere using four separate drilling methods, namely percussive, rotary with water flushing, rotary with air flushing, and rotary with no flushing media. The purpose of these tests is to establish standards of comparison for future drilling tests to be performed in a simulated lunar environment. Data recorded for each test include penetration rate, bit temperature, rpm, and power consumption. Because the percussive drill produces a hole larger than the rotary diamond drill (1-1/8-inch vs 1/2-inch), the energy per unit volume was determined for each system. Figure 1 compares rotary diamond core drilling with and without water flushing with the percussive core drilling method. Figure 2 shows the effect of increased thrust on bit temperature for percussive drilling and rotary drilling without water flushing.

A preliminary analysis of the data indicates that in flood basalt the percussive system shows a higher power consumption per unit volume. The usefulness of the rotary system appears to be largely dependent on the life of the diamond bit.

Tests are now underway to establish approximate bit life figures in the various simulated lunar materials. Preliminary experiments show that

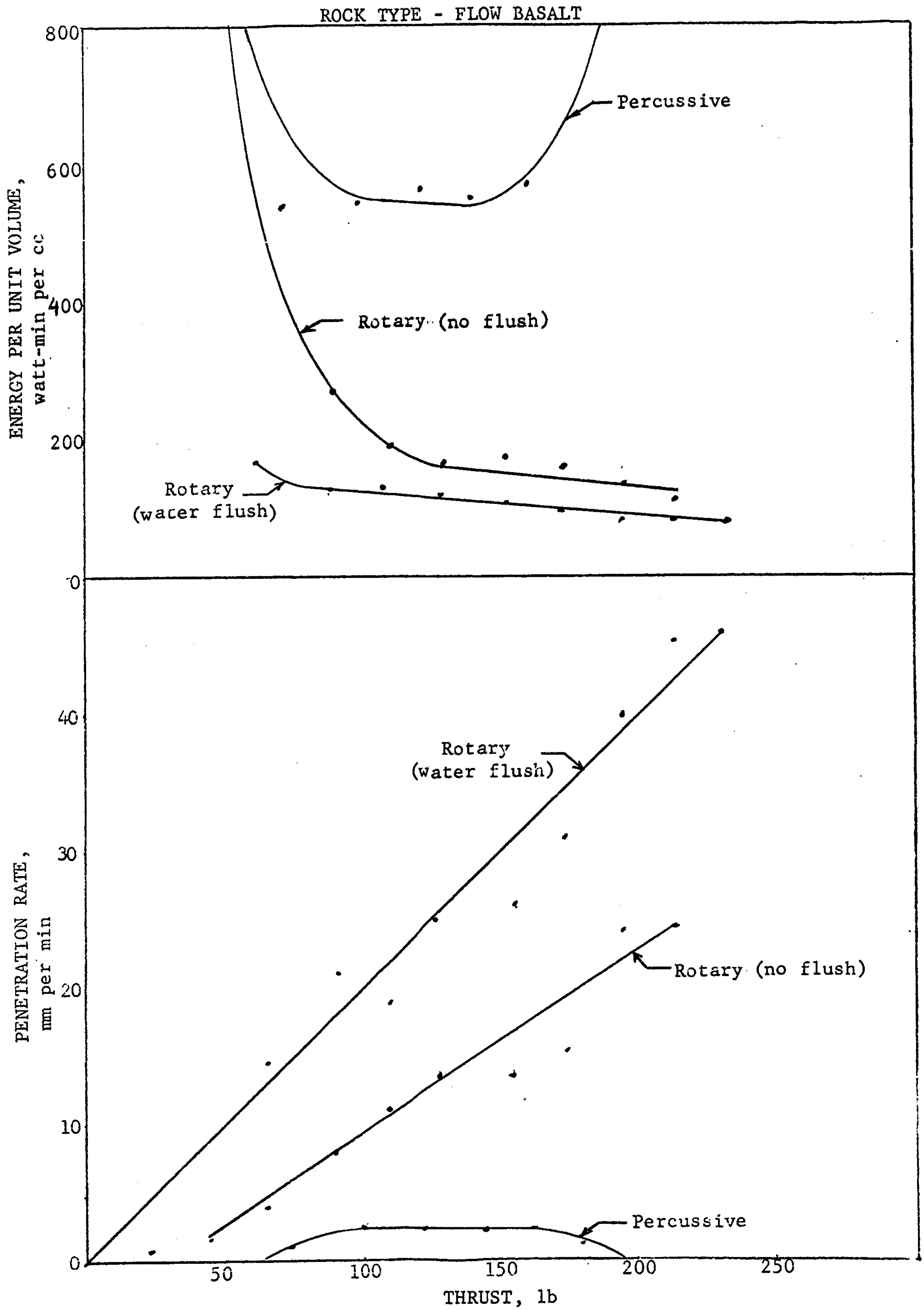

FIGURE 1. - Energy per Unit Volume and Penetration Rate vs Thrust for Flow Basalt

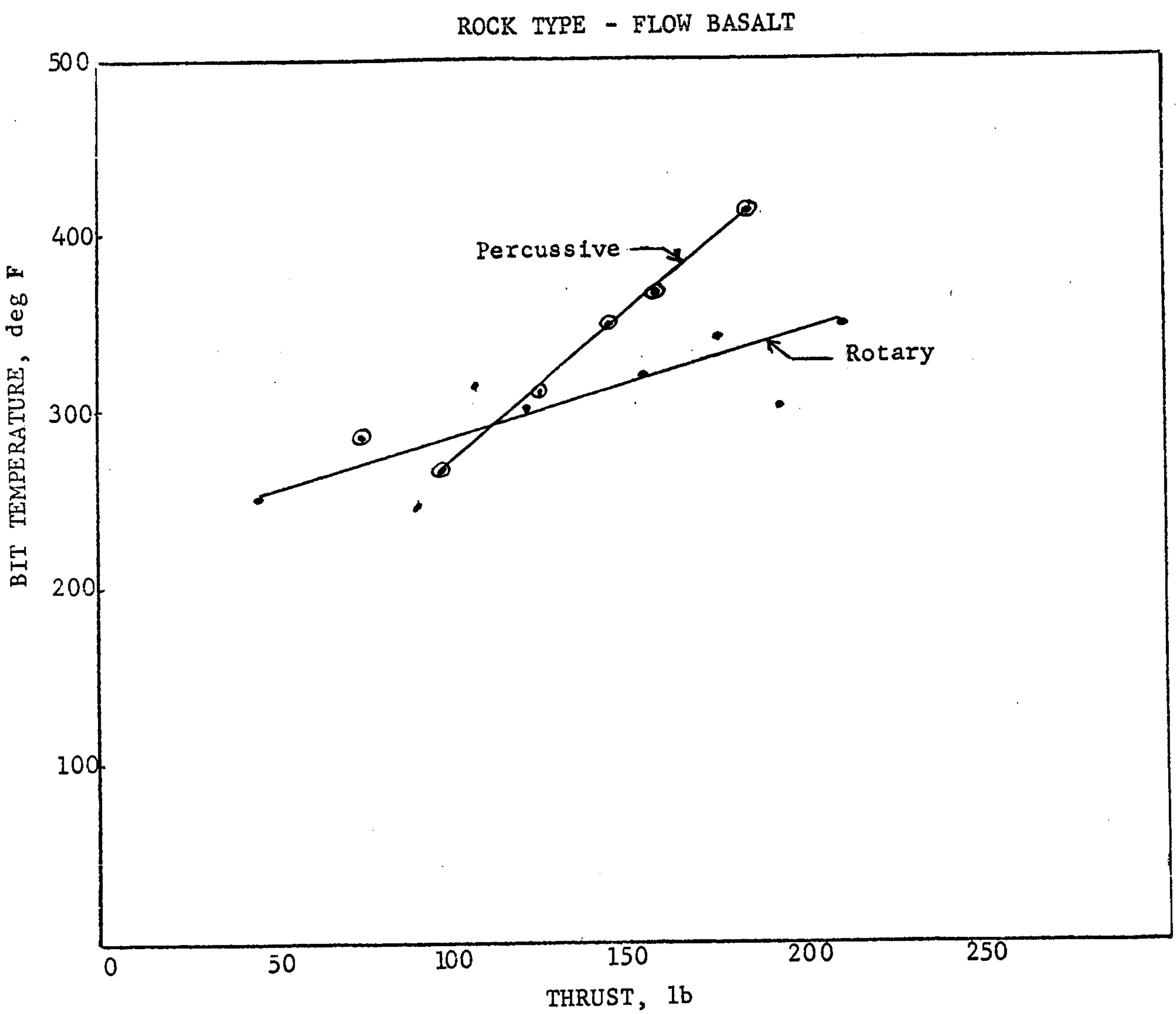

FIGURE 2. - Bit Temperature vs Thrust for Flow Basalt.

if an efficient method of cuttings removal can be designed, the bit life can be greatly increased.

Investigation of experimental techniques for determining the effect of ultrahigh vacuum on drilling parameters was begun. Vacuum chamber facilities at our Center will probably be satisfactory for some experimental work. However, part of the required tests are likely to call for a somewhat larger chamber and faster pumping speed than we can provide. We are exploring with Hoyt Weathers and Stanley Fields at NASA's Marshall Space Flight Center, Huntsville, Ala., the possibility of cooperative experiments using their recently expanded vacuum chamber facilities.

Status of Manuscripts

Cuttings Removal and Bit Cooling in Lunar Drilling, by James Paone, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Effect of lunar environment on behavior of fine particles
Investigator: David E. Nicholson, Project Leader
Location: Spokane Mining Research Laboratory
Spokane, Washington
Date begun: April 1966 To be completed: March 1969

Personnel: David E. Nicholson, Mining Methods Research Engineer
William R. Wayment, Mining Methods Research Engineer
Dennis J. Kelsh, Physical Chemist
Daniel J. Larson, Research Chemist

PROGRESS REPORT

Objective

Extend current studies of fine particle behavior in mine backfill applications to include lunar environment. Measure such properties as density of packing, repose or friction angles, and rates of flow through orifices or channels. This work will be correlated with the study of electrostatic properties of granular particles being conducted at College Park, Md.

Progress During the Third Quarter

A course in vacuum technology at the University of California in Los Angeles was attended during the quarter to assist project personnel in deciding their best approach to lunar vacuum simulation. A memorandum-report detailing various aspects of the course as they affect vacuum testing of fine particles was prepared and submitted to the core group at the Twin Cities Center. It now appears unlikely that we will attempt to obtain lunar vacuum simulation facilities here at Spokane under the present funding limitations of the program. The alternative of having the tests that we need to make in lunar vacuum performed elsewhere will be pursued.

The movement of project equipment from the physical chemistry laboratory at Gonzaga University was completed and all equipment was installed and is now operating in our new laboratory space. A series of horizontal capillarity tests were completed, and vertical capillarity, permeability, and sedimentation volume tests were in progress, on the sized silica fractions.

A full-time research chemist joined the staff of our laboratory at the end of February replacing our part-time university student chemist. He is scheduled to work primarily on the fine particles project, so that property measurement work should accelerate significantly in the future.

Status of Manuscripts

Behavior of Fine Particles, by David E. Nicholson, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Support for underground lunar shelter
Investigator: Ernest L. Corp, Project Leader
Location: Spokane Mining Research Laboratory
Spokane, Washington
Date begun: April 1966 To be completed: March 1969

Personnel: Ernest L. Corp, Mining Methods Research Engineer
Robert C. Bates, Mining Methods Research Engineer

PROGRESS REPORT

Objective

The ultimate objective of this project is to advance the ground support technology needed to carry on extraterrestrial mining in support of space missions. The immediate objectives are: (1) To define the problems which will be encountered in designing a lunar ground support or environmental shelter system; (2) to investigate possible materials (indigenous and transported) which can fulfill the requirements for utilization in a support or environmental shelter system; (3) to formulate design concepts for support systems utilizing the most favorable materials.

Progress During the Third Quarter

The background study on the physical and environmental characteristics of the lunar surface, and on suitable ground support materials has continued on a full-time basis during the quarter. This background study has revealed a number of materials and techniques which might be adaptable to lunar shelter construction.

The majority of research in the area of aerospace structures has been done on the expandable, self-rigidizing type of structure. Typical materials used in these structures are inflatable fabrics made of fiberglass or various plastics, which are rigidized by the dehydration of gelatin solutions or by any one of numerous foaming reactions. Although these materials and construction techniques can offer realistic and immediate solutions to the problem of lunar shelter design (surface or underground), these solutions should be considered only temporary as far as the extraterrestrial mining program is concerned. First of all, the use of such structures as a sole source of shelter on or near the lunar surface will not provide the degree of radiation or meteorite protection required for permanent inhabitance. Secondly, if these structures were at a depth sufficient to provide this protection, the high cost of transportation from Earth would not coincide with the self-sufficiency goals of the mining research program. In addition, extensive research already underway by other investigators would make additional contributions from this project insignificant.

For these reasons, it is felt that future efforts should be concentrated on shelter construction techniques using materials believed to be indigenous to the lunar surface. Such confinement of the project's objectives would eliminate any work on organic materials, and concentrate all research on the various inorganic construction materials which can be derived from lunar rocks and minerals.

One example of such a material is sodium or potassium silicate found in the serpentines, chlorites, micas, and zeolites. Sodium or potassium silicate is commonly produced by reacting silica sand with appropriate alkalies, resulting in a viscous liquid in solution with water. These silicates might be produced as a byproduct in the recovery of water from lunar rocks. The silicate solution when subjected to either heat or vacuum will produce a glass-like foam with properties similar to the plastic foams. If the foam is subjected to additional heating it will form fused glass. The properties of the silicates, therefore, make them ideal for producing foam materials for lunar shelter construction, or sealants for natural and excavated underground openings. As discussed in last quarter's status report, one of the main considerations in using an underground opening for life support is to have a suitable sealant for maintaining the required internal pressurization. In most cases the problem of providing actual ground support will be only a secondary consideration. Therefore, the development of a sealant material applied by a spray-on technique in a low-pressure environment would be a significant contribution to lunar underground shelter technology.

In addition to the silicates, sulfur may also be an indigenous material suitable for lunar construction. Besides its adaptability to foaming, it could also serve as a sealant or a cementing matrix for some easily obtained lunar aggregate.

A third technique worthy of consideration for sealing surfaces of underground openings is a plasma gun. Using this technique, the surface of the opening can either be fused in place or various materials can be introduced to produce a plasma spray coating.

In February the project leader made a visit to Southwest Research Institute in San Antonio, Tex. Southwest personnel are currently conducting projects on both sodium silicate and sulfur as construction materials for a variety of applications. During this visit, a demonstration was given to show the production of sodium silicate foam by subjecting the solution to either a vacuum or a micro-wave heating source. Sustained exposure of the foam to micro-wave heating resulted in the production of fused silica. The high efficiency and extreme versatility of a micro-wave heating unit makes it a worthwhile consideration where heat is required for producing or curing a lunar construction material.

Also in February, the project leader took a five-day course in vacuum technology at the University of California in Los Angeles. A memorandum-report describing the highlights of the course and its effect on project goals was sent to the core group at the Twin Cities Center. The complexity and high cost of vacuum or space simulation testing has produced some skepticism on the part of project personnel as to whether the current project scope will warrant work in high vacuum testing. Since this matter must be given further consideration, plans for purchase of a vacuum chamber will be deleted from the current fiscal year work plans.

Status of Manuscripts

Support for Underground Lunar Shelter, by Ernest L. Corp, was presented at the NASA review meeting at the Twin Cities November 30.

Task title:	(1) Effect of vacuum on explosive properties
	(2) Effect of micrometeoroid bombardment on explosives
	(3) Explosive blast effects in lunar environment
Investigator:	Frank C. Gibson, Project Coordinator, Explosives Physics
Location:	Explosives Research Center
	Pittsburgh, Pennsylvania
Date begun:	July 1966 To be completed: June 1969

Personnel:	Frank C. Gibson, Supervisory Research Physicist
	Richard W. Watson, Research Physicist
	J. Edmund Hay, Research Physicist
	Samuel R. Harris, Research Chemist
	Charles R. Summers, Research Physicist
	William F. Donaldson, Research Physicist
	Elva M. Guastini, Explosives Equipment Operator

PROGRESS REPORT

Objective

To develop a body of knowledge relevant to the use of chemical high explosives under lunar environment. Immediate goals are to determine the hazards associated with the storage, handling, and use of explosives in an environment characterized by high vacuum, extreme temperature cycling and a flux of small hypervelocity particles, and to establish techniques for minimizing these hazards.

Progress During the Third Quarter

The investigation of the effect of a moderate vacuum on the relative sensitivity of two granular explosives at ambient temperature was completed. Both tetryl and RDX at a packing density of about one g/cm^3 were subjected to modified card-gap test evaluations; this method was chosen since the preliminary experiments that were conducted to determine the changes in sensitivity by measurement of failure diameters in conical charges were not completely satisfactory. The continuous detonation-velocity probe system which is successfully employed at ambient pressures to determine velocity decay and detonation cutoff produced anomalous results when used in a vacuum. The Bruceton up-and-down method was used to determine an estimate of the mean gap length, i.e., the length of the plastic barrier used to attenuate the shock delivered by an explosive donor, for 50 percent failures after some preliminary experiments had been used to determine the appropriate instrumentation and the approximate gap value. The charge arrangement illustrated in figure 1 was employed.

The explosive was contained in 1/16-inch walled plastic cylinders, one inch in diameter and five inches long; the walls were perforated and lined with paper to improve the conductance to gas flow. Each charge used an expendable resistive pressure gage as a reaction indicator. The gage was decoupled from the explosives column by a

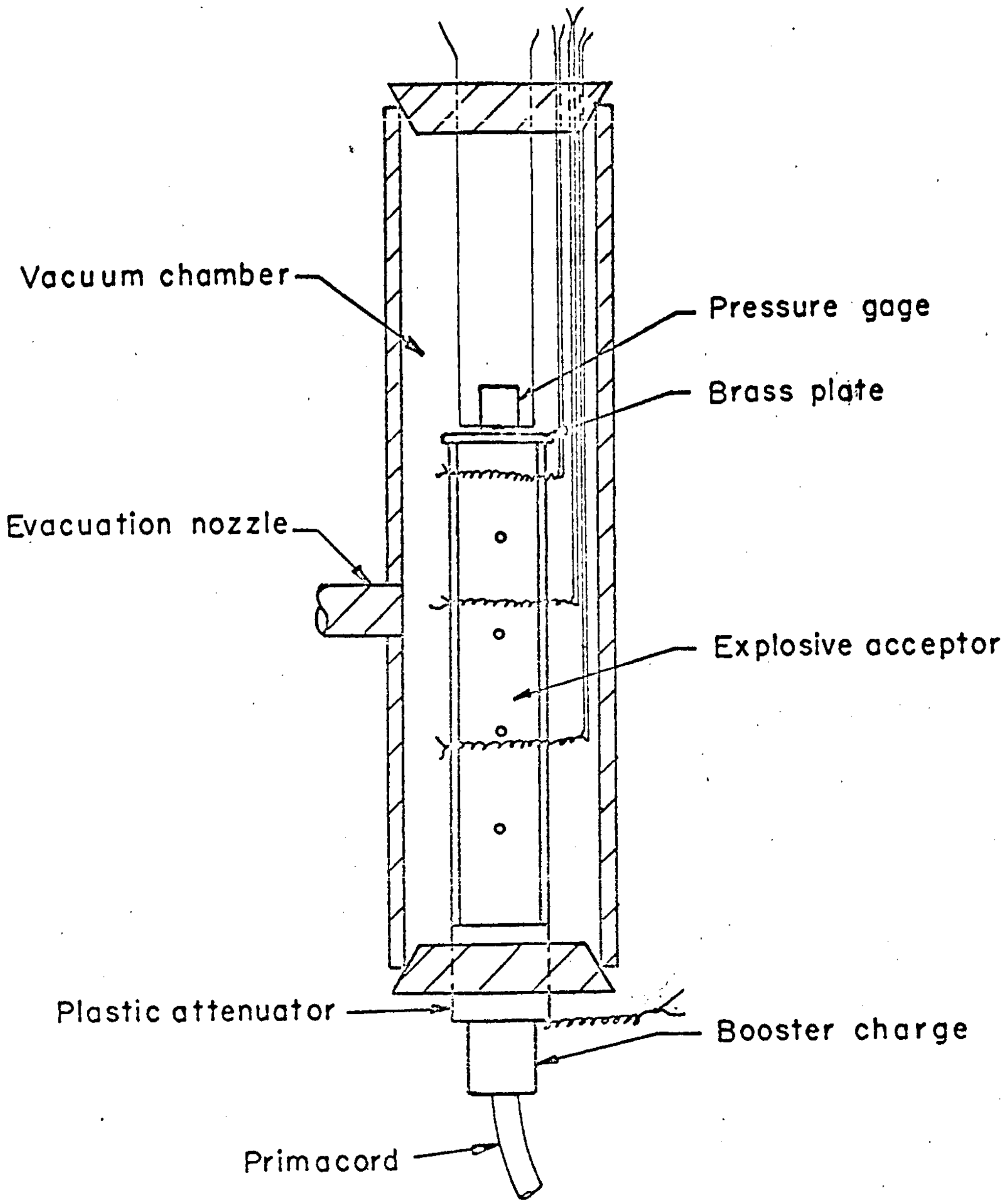

FIGURE 1. - Arrangement used in modified card gap.

brass shock-buffer plate to reduce the pressure incident on the gate to an interpretable level; ionization probes for oscilloscope and counter-chronograph determinations of incremental velocities were installed. The attenuator passed through a rubber stopper that formed one end-seal for the tubular vacuum chamber, the other end being sealed with a similar stopper through which the wire leads extended. A nozzle was attached to the side of the cylinder for evacuation.

In most cases the full assembly was used both at ambient and reduced pressures; however, in all cases the attenuator/stopper arrangement was employed to compensate for possible effects of the rubber surround on the attenuator. The performance of the detonator-explosive booster assembly is not affected since these components are at atmospheric pressure in both cases.

Recognizing the possibility of ambiguous interpretation of the electronic records, six framing camera shots were made to confirm the results; three each were made on tetryl and RDX, two at atmospheric pressure and one in vacuum.

In both the instrumented and photographic trials a vacuum of about 10 microns was employed. The detonation induction delay time and distance into the charge at which a stable rate resulted were approximated from the probe records and from the position of lateral breakout of the detonation products in the framing camera records.

The analysis of the framing records, which are in agreement with the instrumented card-gap test results, shows a basic difference between the two explosives as follows:

Material	Tetryl		RDX	
Density	$\rho_o = 1.0$ g/cm^3		$\rho_o = 1.1$ g/cm^3	
Pressure	Ambient	Vacuum	Ambient	Vacuum
τ (μsec)	28	36	8	10
d (cm)	5.8	5.8	2.22	1.44
$\bar{v}$ (mm/μsec)	2.0	1.6	2.8	1.4

τ is a measure of the time to reaction, d is the distance into the charge from the barrier-explosive interface to the breakout of detonation, and $\bar{v}$ is the average velocity of the initial reaction wave.

In addition, there is a trend indicating that the short exposure to vacuum has increased the induction time which may reflect a reduction in the reactivity of the material. While these experiments are conducted under conditions of marginal initiation and in charges having great porosity, it is possible that long-time exposure of leaky encapsulated charges in a lunar environment may in turn result in degradation of explosive performance. The basic sensitivity as determined from the card-gap analysis showed no significant change between the ambient and evacuated charges of the same material but the order of sensitivity of the tetryl and RDX are reversed with respect to sensitivity at high leading densities, e.g., 1.6 g/cm^3. Gap values for tetryl at ambient and vacuum are 1.34 and 1.35 inches and for RDX at ambient and vacuum 0.95 and 0.94 inch, respectively. The small differences observed for each explosive are probably not significant. These results are based on the "go-no go" propagation of detonation in charges having a length to diameter ratio of about five. A current attempt is being made to determine whether this trend will be continued in pressed charges of a military explosive material, HMX, which is a homologue of RDX that possesses vacuum stability qualities nearest to hexanitrostilbene (HNS), which is the candidate explosive for space use.

In addition to the experiments on HMX, it is planned to start temperature cycling both for the shooting program as well as in a crystallographic analysis of structural changes using X-ray diffraction techniques that will include samples of the HNS with varying amounts of the teflon that is used as a binder. Presently, the candidate composition for lunar use is a 90/10 mixture of HNS and teflon.

Status of Manuscripts

Use of Explosives on the Moon, by Frank C. Gibson and J. Edmund Hay, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Volcanism and ore genesis as related to lunar mining
Investigator: Rolland L. Blake, Project Coordinator
Location: Twin Cities Metallurgy Research Center
Minneapolis-St. Paul, Minnesota
Date begun: June 1966 To be completed: May 1967

Personnel: Rolland L. Blake, Research Geologist
Others as assigned

PROGRESS REPORT

Objective

Study the genesis of ore deposits and the occurrence of minerals associated with volcanic activity here on Earth. Study the effects of the lunar environment and other environments on mineralization and ore genesis. Bring together the pertinent information found in the literature on these subjects and define those specific areas where additional work is needed.

Progress During the Third Quarter

The literature search was continued on the genesis of ore deposits and mineral associations of volcanic origin. Information is being collected under the categories outlined below to facilitate preparation of a report summarizing the findings.

Volcanic Mineralization and Ore Genesis

Earth Volcanism

Extrusive rocks	Intrusive rocks
Minerals	Ores
Processes	Alteration
Volcanic features and forms	

Extraterrestrial Considerations

Environmental effects
Utilization of rocks and minerals

During the California-Oregon field trip, the task objectives were discussed with scientists engaged in research on simulated lunar materials. Dr. Jack Green of the Douglas Advanced Research Laboratories, Huntington Beach, Calif., offered his assistance to the task. He is an expert on volcanic calderas, large depressions formed by collapse of surface volcanic rocks undermined by the outpouring of large volumes of lava. During the quarter, arrangements were completed to procure a report from Dr. Green on caldera mineralization, thereby reducing the amount of literature to be reviewed by the task investigator. The report will summarize current pertinent literature information on calderas, especially that dealing with origin and occurrence of mineral sublimates. It will include a bibliography and will cover such aspects as processes operating to form calderas, caldera location with respect to volcanic and tectonic features of the Earth's crust, and caldera size ranges with a few examples. Dr. Green may speculate on the effect of the lunar environment on caldera processes and products, on utilization of caldera material expected on the lunar surface, and on gaps in our knowledge of calderas and their deposits that suggest areas requiring further study. The report is to be submitted before May 1, 1967.

Status of Manuscripts

Volcanism and Ore Genesis, by Rolland L. Blake, was presented at the NASA review meeting at the Twin Cities November 30.

Calderas as Related to Lunar Exploration, A Survey Report, by Jack Green, to be obtained from Douglas Advanced Research Laboratories by May 1.

Task title:	(1) Reduction of silicates with carbon (2) Reduction of silicates in plasma torch	
Investigator:	Sanaa E. Khalafalla, Project Coordinator	
Location:	Twin Cities Metallurgy Research Center Minneapolis-St. Paul, Minnesota	
Date begun:	June 1966	To be completed: May 1969
Personnel:	Sanaa E. Khalafalla, Supervisory Research Chemist Larry A. Haas, Research Chemist	

PROGRESS REPORT

Objective

To obtain fundamental information on the effect of reaction parameters on the carbothermic reduction of silica at subatmospheric pressure. This is one phase of the multidisciplinary effort to extract oxygen from possible lunar raw materials. Also, the feasibility of reducing silicates with activated hydrogen in a plasma torch will be studied. Experiments are being systematically designed to clearly delineate the variables which would produce the optimum experimental environment for obtaining the highest yields at the lowest processing cost. The ultimate tangible results will consist of mechanistic theories and kinetic equations expressing the reaction rate as a function of temperature, pressure, reactants' ratio and particle size.

Progress During the Third Quarter

Research has been directed to obtain a baseline which can be used as a reference in comparing the effects of various physicochemical variables on the carbothermic reduction of silica. In the preceding quarters, this solid-solid reaction was investigated with finely divided reactants of minus 400-mesh graphite and minus 200- plus 270-mesh silica. With these fine particles it was difficult to retain the entire charge in the reacting chamber at temperatures above 1,300°C. This was attributed to the formation of CO with high evolution flow rates which expelled some of the unreacted sample. To avoid the blowout problem, attempts were made to briquet the loose powders at 50 tons per square inch in one trial and to agglomerate the powders with 2 percent aqueous dextrose in another trial. Both methods were found unsuccessful in eliminating the sample blowout. This problem was successfully resolved through the use of larger particles along with a molybdenum baffling system fitted with a quartz wool filter.

The immediate goal of the work during the past quarter was to investigate the effect of temperature and reactants' ratio on the silica reduction rate with carbon. The dependence of the rate on these variables is of paramount importance in elucidating the reaction mechanism. The experimental results should also aid in predicting the optimum conditions for extraction of oxygen from silica-bearing materials.

The temperature dependence of the reaction rate was determined with minus 80- plus 120-mesh SiO_2 and minus 100- plus 200-mesh graphite. The reduction rate is approximately proportional to the weight loss, and was calculated from the material balances of both silica and carbon. Comparison of the observed and calculated weight loss indicated a reasonable agreement, as shown in table 1. The calculated weight loss was determined assuming the only gaseous products of reaction are CO and Si. Support for this assumption was obtained from the approximate equality of the number of atoms of oxygen and carbon lost. The following two reactions are probable in vacuum and at temperatures above 1,300°C:

$$SiO_2 + C \longrightarrow SiO \uparrow + CO \uparrow \qquad (1)$$

$$SiO_2 + 2C \longrightarrow Si \uparrow + 2CO \uparrow. \qquad (2)$$

According to equation 1, the atomic ratios of oxygen to carbon in the gas phase is two, while according to equation 2 this ratio is one. The results in table 1 indicate that this ratio is closer to one at the high temperatures and high silica contents. Deviations at the low silica or high carbon ratios can be attributed to the carbon gasification through oxidation with residual furnace air. This was tentatively verified by duplicating two tests at different furnace leak rates. In test No. 1, the leak was more than three times larger than in test No. 2 and accordingly, more weight was lost in the former tests. A blank test for the gasification of carbon with residual oxygen indicated a weight loss of 0.14 percent per hour at 1,400°C. Hopefully, the air leakage problem will be overcome through the acquisition of a new vacuum furnace.

Data for the temperature effect on the reaction rate are given in table 1. In subatmospheric conditions the carbothermic reduction of silica exhibits a sharp rate increase above 1,400°C, as shown in figure 1.

The dependence of reaction rate on the reactants' molar ratio was also determined. It was difficult to correlate the experimental data as the reaction appeared to be slightly dependent on the amount and depth of the sample. It was also difficult to vary the molar ratio of the reactants without at the same time changing the volume of the charge. The results of tests No. 17 and 18 in table 1 indicate that doubling the quantity of each reactant almost doubles the amount of oxygen extracted. The sample concentration profile of test No. 18 also showed that more carbon was utilized in the upper portions of the charge.

Investigations of the dependence of the reaction rate on the molar ratio of silica to graphite in the range of 0.5 to 8 was carried out by using 0.2 moles of silica and variable amounts of graphite. For molar ratios in the range from 0.125 to 2, the tests were performed using 0.2 moles of graphite and variable amounts of silica. A plot of the percent oxygen extracted in five hours against the molar ratio of silica to graphite shows a maximum reaction rate at a molar ratio of about four (figure 2). When the units of the abscissa of figure 2 were changed

TABLE 1. - Experimental results of the reaction of -80, + 120-mesh silica with -100, + 200-graphite in vacuum (< 10^{-2} torr)

Test No.	Silica, %	SiO_2/C molar ratio	Charge, grams	Temp., ° C	Carbon oxidized, moles	Oxygen extracted, moles	Gaseous atomic ratio, O/C	Total oxygen extracted, percent	Five hour weight loss, percent Calculated[1]	Five hour weight loss, percent Observed
1				1350	0.028	0.008	0.6	4	6.6	5.3
2				1350	0.016	0.003	0.4	1.5	3.9	2.8
3				1375	0.024	0.005	0.4	2.5	5.8	4.2
4	83.3	1.0	14.4	1395	0.034	0.014	0.8	7	8.1	7.5
5				1405	0.036	0.014	0.8	7	8.2	7.2
6				1410	0.041	0.020	1.0	10	9.1	9.0
7				1425	0.065	0.029	0.9	14.5	13.3	13.7
8				1445	0.103	0.050	1.0	25	22.2	21.7
9	38.5	0.125	3.9		0.003	0.000	- -	0	2.8	1.8
10	55.6	0.25	5.4		0.007	0.001	0.3	0.5	4.5	2.4
11	71.4	0.50	8.4		0.015	0.001	0.2	0.5	5.9	3.4
12	71.4	0.50	16.8		0.031	0.008	0.5	4	6.3	4.8
13	71.4	0.50	16.8		0.028	0.007	0.6	3.5	5.9	4.8
14	83.3	1.00	14.4		0.033	0.010	0.6	5	7.9	6.4
15	83.3	1.00	14.4	1400	0.036	0.012	0.6	6	8.6	7.2
16	83.3	1.00	14.4		0.037	0.014	0.8	7	8.4	7.2
17	90.9	2.00	13.2		0.033	0.015	0.9	7.5	8.5	7.7
18	90.9	2.00	26.4		0.046	0.028	1.2	7	7.0	7.1
19	93.0	3.00	12.9		0.030	0.014	1.0	7	8.0	7.7
20	95.2	4.00	12.6		0.032	0.020	1.2	10	10.6	11.6
21	96.2	5.00	12.5		0.035					21.8
22	97.6	8.00	12.3		0.023	0.005	0.4	2.5	8.7	7.2

1/ Weight loss was calculated on the basis that the only gaseous products of the reaction are Si and CO.

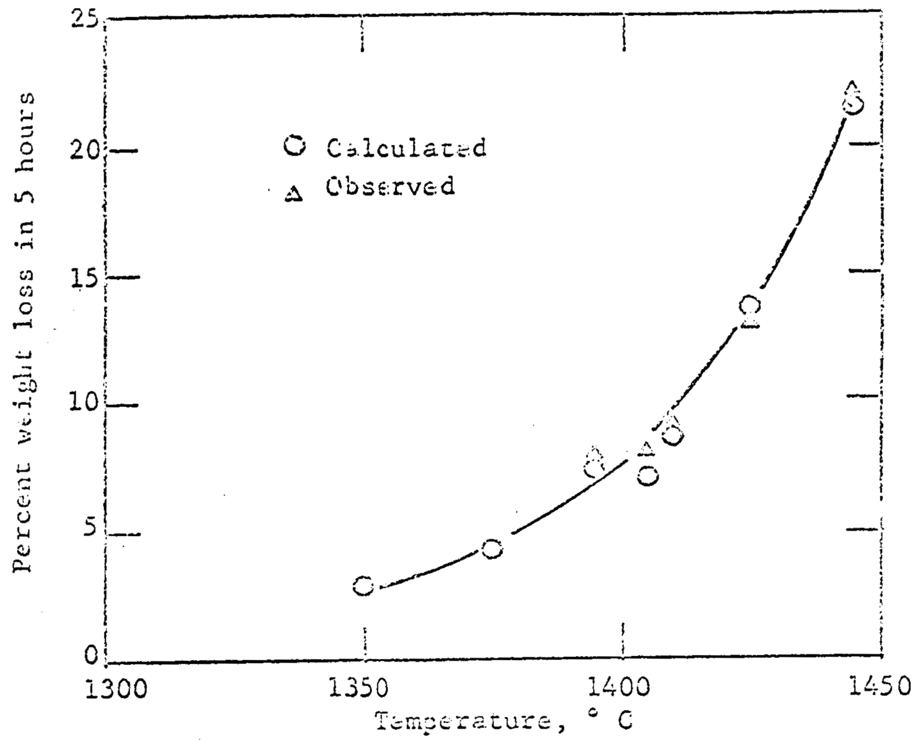

FIGURE 1. - Temperature Dependence of the Carbothermic Reduction of Silica in Vacuum (< 10^{-2} torr).

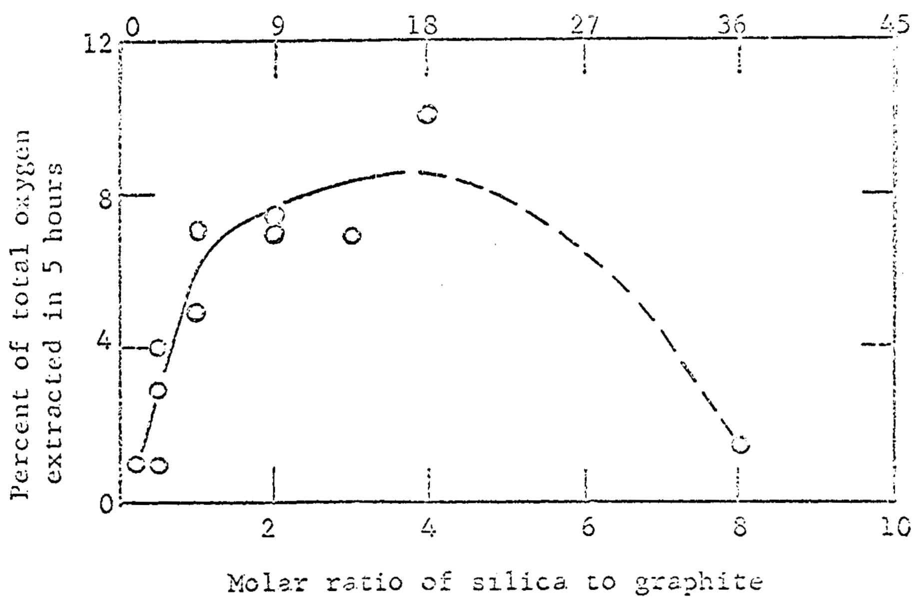

FIGURE 2. - Reduction Rate Dependence on the Molar Ratio of the Reactants.

from molar ratio to particle ratio, the maximum reaction rate occurred when the particles of silica outnumbered those of graphite by a factor of about 18. The molar to particle ratio conversion was calculated through the average particle size and density of each constituent.

In the next quarter, the reactant particle ratio studies will be continued. The optimum particle ratio will be confirmed by varying the size of one of the reactants independently of the other. Research on the feasibility of reducing silicates with activated hydrogen in a plasma torch has been delayed due to loss of two professional employees.

Status of Manuscripts

Reduction of Silicates with Carbon, by Larry A. Haas and Sanaa E. Khalafalla, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Magnetic and electrostatic properties of minerals in a vacuum
Investigator: Foster Fraas, Project Leader
Location: College Park Metallurgy Research Center
College Park, Maryland
Date begun: June 1966 To be completed: May 1969

Personnel: Ray A. Heindl, Supervisory Chemical Research Engineer
Foster Fraas, Research Metallurgist

PROGRESS REPORT

Objective

Study adsorption and contact electrification in a vacuum and determine their effect on the separability of nonconducting minerals.

Progress During the Third Quarter

The objectives of research during the third quarter were to assemble equipment and conduct preliminary measurements as to the relative extent of interfering and useful electrification effects for particle separation and particle flow.

Vibrating electrifiers have been constructed and tested for vibration drive transmitted by rods through vacuum seals, instead of by the usual method of an electromagnetic drive close to the vibrator.

Plans were drawn for the construction of a feed through for 120 cycles per second vibration transmission to the vibrating electrifier, using a stainless steel bellows. A feed through of this type could not be found on the market.

Although original plans were for the initial use of a pyrex bell jar, this has been changed to stainless steel to avoid permeation of atmospheric helium through the pyrex glass.

Progress has been retarded not only by the delay in equipment delivery, but also by the necessity for considerable design and construction on the vacuum equipment itself. Instead of purchasing a complete vacuum unit, the pump, base plate, bell jar, and hoisting equipment were of necessity purchased from different manufacturers and require additional construction for assembly.

Status of Manuscripts

Electrostatic Properties in Vacuum, by Foster Fraas, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Biological production of sulfuric acid
Investigator: Joseph A. Sutton, Project Leader
Location: College Park Metallurgy Research Center
College Park, Maryland
Date begun: June 1966 To be completed: May 1967

Personnel: Ray A. Heindl, Supervisory Chemical Research Engineer
Joseph A. Sutton, Research Chemist
John D. Corrick, Research Chemist
Jerry M. Carosella, Microbiologist

PROGRESS REPORT

Objective

Establish the limiting environmental conditions for the survival of bacteria of the genus thiobacillus. Determine the rate of sulfuric acid production within these limits. Conduct a literature survey and visit such laboratories as may be necessary to establish the state of the art in the use of bacteria in any stage of a life support system in an extraterrestrial environment.

Progress During the Third Quarter

The objectives for the quarter were to continue the determination of the effect of temperature variations on microbial growth and acid production at different pressures. Any combination of temperature and pressure that shows promise will be investigated in more detail.

The results obtained to date are summarized in tables 1 and 2.

TABLE 1. - Effect of variations in temperature and pressure on acid production

Temperature °C	Acid produced, percent of control		
	2 psia	14.7 psia (control)	30 psia
30	50	100	100
25	70	100	85
20	80	100	35

TABLE 2. - Effect of variations in temperature and pressure on cell reproduction

Temperature °C	Cells per milliliter, percent of control		
	2 psia	14.7 psia (control)	30 psia
30	20	100	65
25	50	100	60
20	90	100	20

The results obtained at 20°C confirmed the trend shown in previous experiments in that as the temperature was lowered the acid produced and cell count per milliliter under partial vacuum approached the control, while at a pressure of 30 psia, the opposite effect was observed.

Status of Manuscripts

Biological Production of Sulfuric Acid, by Joseph A. Sutton, was presented at the NASA review meeting at the Twin Cities November 30.

Task title:	Electrowinning of oxygen from silicate rocks
Investigator:	Donald G. Kesterke, Project Leader
Location:	Reno Metallurgy Research Center Reno, Nevada
Date begun:	June 1966 To be completed: May 1969
Personnel:	Don H. Baker, Jr., Supervisory Research Metallurgist Donald G. Kesterke, Research Extractive Metallurgist Freddy B. Holloway, Physical Science Technician

PROGRESS REPORT

Objective

To determine the feasibility of obtaining elemental oxygen from silicate minerals by electrolytic methods, for use by the Earth inhabitants of the Moon. Emphasis will be directed toward the determination of essential physical and electrochemical properties of silicate and silicate-base melts containing various amounts of halide salts. Complementary investigations will be made to find suitable nonreactive crucible and anode materials for use in silicate melts, or in melts containing halides.

Progress During the Third Quarter

In past work melting characteristics and relative viscosities of various silicate-bearing minerals were determined. It was established that fluxing agents are necessary to lower the solidus temperature and increase the fluidity of the molten silicates. During this quarter experiments were begun to determine whether the rock mixtures plus added fluoride have sufficient conductivity, as indicated by the voltage-amperage relationship, to be used as an electrolyte.

Work was initiated on two mixtures of a basalt plus a pumice-like sinter; the first contained 10 weight-percent LiF and the second 15 percent as fluxing agents. The procedure consisted of immersing two 3/8-inch diameter graphite electrodes, spaced 3 inches apart, a fixed distance into the melt, and applying direct current from a regulated power source.

It was determined that both temperature and fluoride content have a marked effect on the conductivity of the melts. At 1,100°C, neither melt was very conductive. The mixture containing 10 weight-percent LiF required an emf of more than 16 volts to achieve a current strength of 6 amps at 1,200°C and at 1,300°C the same current required about 9 volts. The mixture containing 15 weight-percent LiF was considerably more conductive. Under identical conditions, a current of 6 amps required 8 volts at 1,200°C and only about 4 volts at 1,300°C.

Similar tests will be performed on the basalt-plus-sinter mixture and other silicate mixtures, using CaF_2 as the fluxing agent.

Dr. Henrie, the original investigator for this task, has assumed additional responsibilities as Research Director for the Center. Mr. Kesterke has taken over as investigator with Mr. Baker as his supervisor.

Status of Manuscripts

Electrowinning of Oxygen from Silicates, by Donald G. Kesterke, was presented at the NASA review meeting at the Twin Cities November 30.

Task title:	Stability of hydrous silicates and oxides in lunar environment
Investigator:	Hal J. Kelly, Project Coordinator
Location:	Albany Metallurgy Research Center Albany, Oregon
Date begun:	April 1966 To be completed: March 1968
Personnel:	Hal J. Kelly, Supervisory Ceramic Research Engineer Raymond L. Carpenter, Research Physicist

PROGRESS REPORT

Objective

The long-range objective is the determination of the energy requirements for dissociating silicate and oxide minerals to recover oxygen and/or water. The immediate objective is to investigate the stability under high vacuum and elevated temperature of some silicate and oxide minerals employing differential thermal analysis (DTA) and thermogravimetric analysis (TGA).

Progress During the Third Quarter

The DTA equipment has been modified. A new sample chamber has been made. It consists of a heavy-walled cylindrical nickel heat shield with a cover and base plate. The base plate is supported by the two alumina thermocouple insulators which contain the thermocouples which measure the temperatures of the sample and reference materials. The cylindrical heat shield sits on this base. Nickel crucibles with thermocouple wells in their bases are set in over the insulators so that the beads of the thermocouples are in contact with the nickel crucibles.

A three-wire thermocouple hookup as described by Wendlant, (J. Chem. Edu., v. 40, No. 8, August 1963, p. 429) was used. This has the advantage that the recorder plots the temperature of the standard material rather than the temperature in some area of the furnace removed from the standard as is usually done.

The first tests run with the new setup were run in air without crucibles on the thermocouples. These tests showed that the baseline was stable and did not drift from the zero position by more than 2°C. Test runs were also made on kaolinite and quartz in air and in vacuum. Satisfactory curves were obtained for these materials although there was more baseline drift in the vacuum runs than on the runs in air.

For kaolinite run in vacuum the endothermic peak, which is caused by the loss of water by the clay, was at 565°C, which is 50° lower than the peak temperature for this material run in air. The area under the curve was smaller for the vacuum run than for the run in air. The exothermic peak at 970°C, which is due to a change in crystal structure, did not show a shift in temperature. A recorder has been connected to the vacuum gauge so that a record could be made of the pressure changes resulting from outgassing. The pressure time curve for kaolinite indicates that the decomposition could be starting as low as 275°C and that decomposition is essentially over at the time the endothermic peak is reached.

Preliminary calibration runs were made on the equipment in air and in vacuum. Boersma, (J. Am. Ceram. Soc., v. 38, No. 8, 1955, p. 283), states that the area under the DTA curve is equal to the product mass of the sample and the heat of reaction divided by a heat transfer coefficient. This transfer coefficient will be different in air and vacuum. Since the exothermic peak temperature of kaolinite does not change due to vacuum, and since there is no reason to believe the energy of the reaction would change because it is a change in crystal structure, it was used for calibration of the vacuum DTA. This was done in the following way. First, two runs were made with CP $CaCO_3$ in air. This material decomposes at a peak temperature of 985°C. The areas under the curves were measured. The heat of reaction multiplied by the mass of the sample was divided by the area. This gave a calibration factor for air of 2.21 cal/unit area. Next, two runs were made on meta-kaolinite; the area under the curve was measured and the energy transformation calculated using the value of 2.21 cal/unit area. The average value, 39.0 cal/gm, was used to find the calibration factor in vacuum. This was determined as 1.82 cal/unit area. Checks of the calibration factor for air with kaolinite and magnesite indicate that heats of reaction can be calculated to about 10 or 15 percent.

The study of the thermal decomposition of the mica series of minerals has been started and will be continuous during the next quarter.

Status of Manuscripts

Stability of Silicates and Oxides, by Hal J. Kelly, was presented at the NASA review meeting at the Twin Cities November 30.